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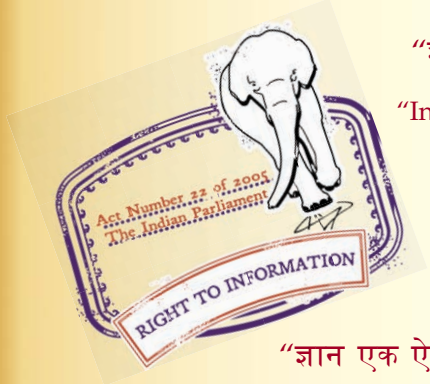

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IS : 539 - 1974
(Reaffirmed 1993)

Indian Standard

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SPECIFICATION FOR NAPHTHALENE

(Second Revision)

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SPECIFICATION FOR NAPHTHALENE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 7 November 1974, after the draft finalized by the Coal Carbonization Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1955. In view of the growing demand by chemical industries for hot-pressed naphthalene, the standard was revised in 1965 and the requirements of the two grades of naphthalene were prescribed in the standard. With the experience of tar distillation plants attached to steel plants, and others, it has now been felt that the requirements of insolubles in benzene and non-volatile matter should also be incorporated in the standard. Accordingly, these requirements are covered in the second revision of the standard.

0.3 In drawing up the second revision, the requirements for insolubles in benzene and non-volatile matter for both the grades of naphthalene have been incorporated. Limits for total sulphur and total nitrogen for hot-pressed naphthalene have also been revised in the light of experience gained by the industry in recent years. This standard which has been drawn with the collaboration of all concerned, is expected to assist the manufacturers in producing and consumers in selecting a material of acceptable quality.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and tests for naphthalene for use as moth-repellant, hides and skin preservative and as a chemical for the production of organic intermediates in the manufacture of dyes, drugs, etc.

2. TERMINOLOGY

2.0 For the purpose of this standard, the definitions given in **2.1** and **2.2** shall apply.

2.1 Repeatability — A quantitative measure of the variability associated with a single operator in a given laboratory obtaining successive repeat results on the same apparatus. It is defined as that difference between two such single results that would only be exceeded in the long run in one case in twenty in the normal and correct operation of the test method.

2.2 Reproducibility — A quantitative measure of the variability associated with operator working in different laboratories each obtaining single result on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run in one case in twenty in the normal and correct operation of the test method.

3. GRADES

3.1 There shall be two grades of naphthalene, namely:

Grade 1 — Naphthalene, pure; and

Grade 2 — Naphthalene, hot-pressed.

4. REQUIREMENTS

4.1 Description

4.1.1 Grade 1 material shall consist of prime, white balls, blocks, flakes or crystalline powder and shall be free from dirt or oily impurities.

4.1.2 Grade 2 material may be obtained by hot-pressing, centrifuging, etc, and is generally known in trade as naphthalene, hot-pressed. It shall be white to light brown in colour and shall consist of lumps, blocks or granules.

4.2 Test for Staining — Grade 1 material shall not produce any stain in contact with woollen fabrics when tested as prescribed below.

4.2.1 Wrap a small quantity of the material tightly in a piece of white serge, and maintain it at 50 ± 1°C for 4 hours. Then examine the piece of serge for stains, if any.

4.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR NAPHTHALENE

Sl. No.	Characteristic	Requirement		Method of Test, Ref to	
		Grade 1	Grade 2	Appendix	Indian Standard
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour	White	When compared in identical glass tubes, the powdered material shall be not darker than a solution of 20 mg of iodine and 40 mg of potassium iodide in 100 ml of water	—	—
ii)	Crystallizing point, °C, *Min*	79·4	78·5	A	—
iii)	Moisture content, percent by mass, *Max*	0·2	0·2	B	—
iv)	Ash, percent by mass, *Max*	0·02	0·02	C	—
v)	Acid wash test	Colouration not darker than No. 3 standard colour described in the test	—	D	—
vi)	Matter insoluble in benzene, percent by mass, *Max*	0·1	0·1	E	—
vii)	Non-volatile matter at 160°C, percent by mass, *Max*	0·1	0·1	F	—
viii)	Total sulphur, percent by mass, *Max*	—	0·4	—	5.1 of IS : 1350 (Part III)-1969*
ix)	Total nitrogen, percent by mass, *Max*	—	0·1	—	IS : 1350 (Part IV/ Sec 2)-1975†

*Methods of test for coal and coke: Part III Determination of sulphur (*first revision*).

†Methods of test for coal and coke: Part IV Ultimate analysis, Sec 2 Determination of nitrogen.

5. PACKING AND MARKING

5.1 Packing — The material shall be packed as agreed to between the purchaser and the supplier.

5.2 Marking — Each container shall be securely closed and shall be marked legibly and indelibly with the following information:

a) Name and grade of the material;

b) Manufacturer's name and his recognized trade-mark, if any;

c) Gross, net and tare mass; and

d) Batch number.

5.2.1 The product may also be marked with Standard mark.

5.3 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6. SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in Appendix G.

7. TEST METHODS

7.1 Tests shall be conducted as prescribed in IS : 1350 (Part III) - 1969*, IS : 1351 - 1959† and Appendices A to F of this standard. References to Appendices and the Indian Standards are given in col 6 and 5 of Table 1 respectively.

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1960‡) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

*Methods of test for coal and coke: Part III Determination of sulphur (*first revision*).

†Methods of test for coal and coke — ultimate analysis.

‡Specification for water, distilled quality (*revised*).

APPENDIX A

[*Table* 1, *Item* (ii)]

DETERMINATION OF CRYSTALLIZING POINT

A-1. APPARATUS

A-1.0 The apparatus of the shape, dimensions and tolerances given in Fig. 1, consists of the following.

A-1.1 Outer Glass Test Tube — serves as an air jacket and is weighted with lead shots or a similar loading material. It is provided with a cork through which the inner tube (*see* **A-1.2**) is held in position.

A-1.2 Inner Glass Test Tube — fitted with a cork which carries a stirrer in the form of a loop of glass with a glass stem and the thermometer placed centrally within the tube and the glass loop. The bottom of the bulb of the thermometer shall be about 10 mm from the bottom of the inner glass test tube. The cork is so fixed that the immersion mark on the thermometer is in level with the top of the cork.

A-1.3 Cooling Bath — 1 000-ml beaker about 150 mm in height. The level of the cooling liquid in the bath shall be at least as high as the level of the sample in the inner tube.

A-1.4 Thermometer — It shall be of the mercury-in-glass type, solid stem and filled with inert gas at suitable pressure. The stem shall be of lead glass or other suitable glass with enamel back. It shall further conform to the following requirements:

Range	65 to 90°C
Graduation	0·1°C
Immersion, mm	100 (from bottom of bulb)
Overall length, mm, *Max*	400
Stem diameter, mm	6 to 7
Bulb shape	Cylindrical
Bulb length, mm	15 ± 5
Bulb diameter, mm	4·5 to 6·0
Length of main scale, mm, *Min*	220
Longer lines at each	0·5 and 1°C
Fully figured at each	2°C

Expansion chamber	Required to allow heating to 110°C
Top finish	Ring
Scale error not to exceed	± 0·4°C
Use before first stability check, hours, *Max*	500

A-1.4.1 Any thermometer of similar range and accuracy may be used.

A-1.4.2 The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

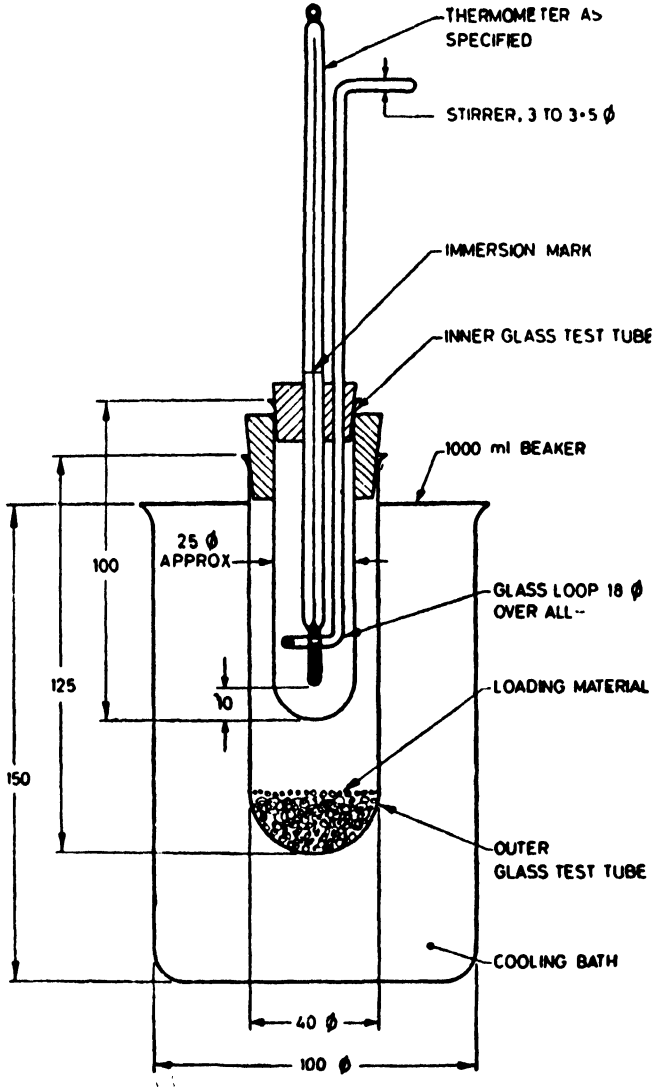
A-2. PROCEDURE

A-2.1 Weigh as quickly as possible about 40 g of the material in a conical flask. Loosely stopper the flask and warm it in boiling water-bath to about 85°C. Mix the molten material thoroughly and pour about 20 g of it into the warmed inner tube of the crystallizing point apparatus. Place the tube in its jacket and assemble the apparatus as shown in Fig. 1 with the bath at 75°C. Discontinue heating the water-bath.

A-2.2 Note the thermometer readings at intervals of half a minute, with continuous and gentle stirring, this operation being so conducted that seed crystal is present as the temperature of the sample falls to that at which crystallization commences. The crystallizing point corresponds to the first five consecutive readings during which the temperature remains constant within 0·05°C.

A-2.2.1 If supercooling takes place, the constant temperature shall be observed immediately after the temperature rise. A temperature rise of 1°C shall be regarded as the maximum allowable.

A-2.2.2 If a constant temperature is not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time interval and a straight line drawn to lie evenly between the first and second and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature rise. The point of intersection shall in this case be taken as the crystallizing point.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF CRYSTALLIZING POINT

APPENDIX B

[*Table* 1, *Item* (iii)]

DETERMINATION OF MOISTURE CONTENT (DEAN AND STARK METHOD)

B-0. GENERAL

B-0.1 Outline of the Method — The material is heated under reflux with an organic solvent which is immiscible with water. The carrier liquid distills into a graduated receiver carrying with it water which then separates to form the lower layer, the excess carrier liquid overflowing from the trap and returning to the still.

B-1. APPARATUS

B-1.1 The apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and the flask shall be interchangeable ground-glass joints. The trap serves to collect and measure the condensed water, and to return the solvent to the flask. The assembly of the apparatus is shown in Fig. 2, and the various components are described below.

B-1.1.1 *Flask* — a 500- to 1 000-ml flask of the shape shown in Fig. 2, made of hard resistance glass, well annealed and as free as possible from striae and similar defects.

B-1.1.2 *Condenser* — a glass water-cooled reflux type condenser, of the design and dimensions shown in Fig. 3. The only mandatory dimensions for the condenser are the external diameters of the inner tube and of the jacket, which shall be 16 to 17 mm and 23 to 25 mm respectively. The joints *A* and *B* shall be neatly finished as shown in Fig. 3, particularly the bore at *B* shall have the minimum disturbance. The shoulder above the cone of joint *D* shall be elongated as shown in Fig. 3 to avoid a sharp re-entrant shape which may restrict the free flow of liquid down the inner wall. The cone shall be extended beyond the length appropriate to the joint *D*, and the lower end ground at an angle of approximately 60° to the axis. The drainage tip shall be at the front of the condenser when the lower water connection is to the left, and the finish shall be either smooth or fire-polished. When inserted into the trap, the tip of the condenser shall be 6 to 7 mm above the surface of the liquid in the trap after distillation conditions have been established. The nominal

dimensions of the joint *D* are given below:

Nominal Dia of Large End of Ground Zone	*Nominal Dia of Small End of Ground Zone*	*Nominal Length of Ground Zone Measured Axially*
mm	mm	mm
18·8	16·2	26

B-1.1.3 *Receivers* — also called the trap, made of hard resistance glass, well-annealed and as free as possible from striae and similar defects, provided with ground-glass joints, with the shape, dimensions and tolerances given in Fig. 4; consisting essentially of the upper chamber, together with the tube and ground joint leading to the flask, and the graduated tube.

B-1.1.3.1 The receivers shall be of 2 ml capacity (*see* Fig. 4). The mandatory dimensions and tolerances for the receiver shall be as given in Table 2.

B-1.1.3.2 The shoulder of the upper chamber of the receiver immediately below the conical joint shall be finished square, as shown in Fig. 4. The graduated portion of the receiver shall be cylindrical throughout its length. The bottom of the graduated tube of the 2-ml receiver shall be sealed, the end of the tube being approximately hemispherical in shape. The graduated scales on the receivers shall be numbered and subdivided as shown in Fig. 4. The graduation marks shall be fine cleanly etched permanent lines of uniform thickness lying in plane at right angles to the axis of the tube. The graduation marks shall be confined to the cylindrical portion of the tube and there shall be no evident irregularity in their spacing. In this receiver the numbered graduation marks shall be carried completely round the tube, the shortest graduation marks shall be carried halfway round the tube, and the graduation marks of intermediate length shall be carried approximately two-thirds of the way round the tube and shall project equally at each end beyond the shortest graduation marks.

B-1.1.3.3 The capacity corresponding to any graduation mark is defined as the volume of water at 27°C, expressed in millilitres, required to fill the graduated portion to that mark at 27°C, the axis of the graduated portion being vertical and the lowest point of the water meniscus being set on the graduation mark.

B-1.1.3.4 The error at any point on the receiver scale, and also the difference between the errors at any two points on the scale, shall not exceed the figures given for the receiver in Table 2.

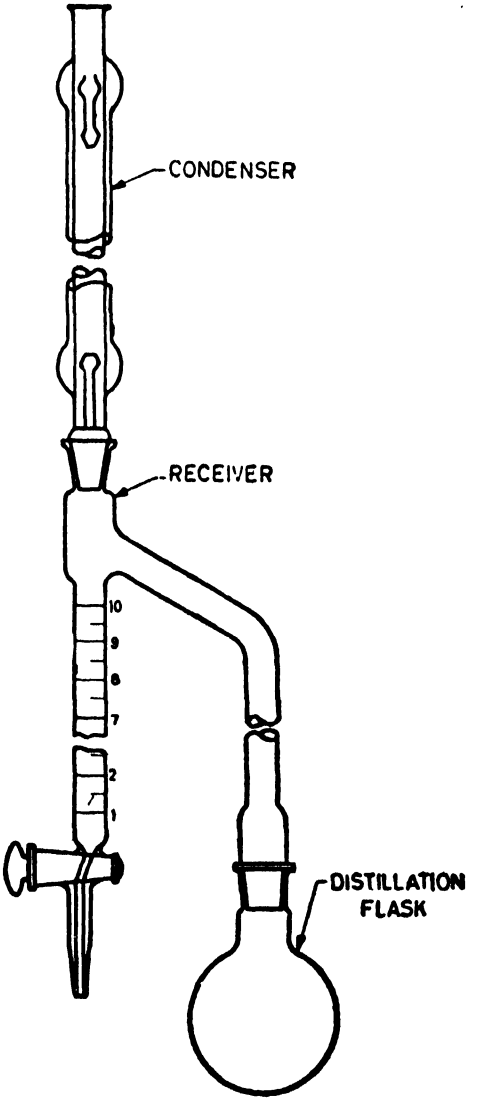
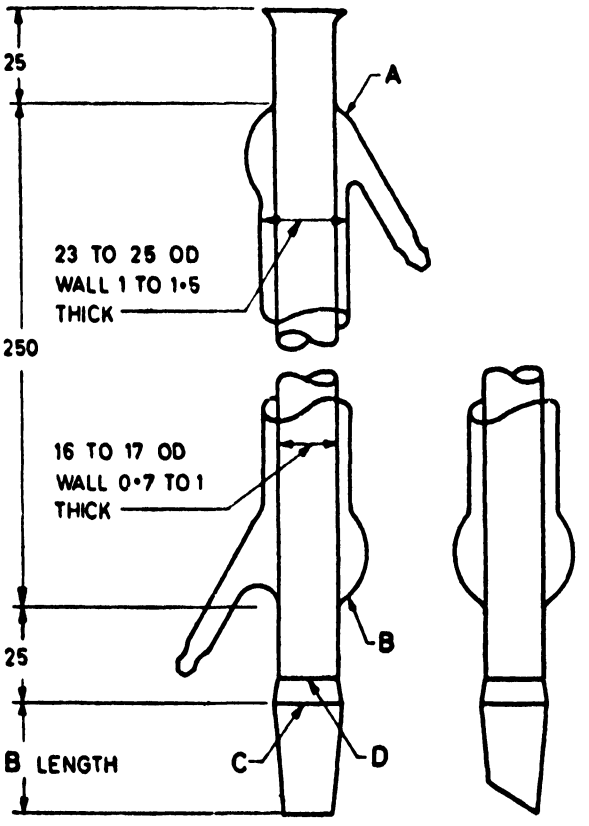


FIG. 2 TYPICAL ASSEMBLY OF DEAN AND STARK APPARATUS



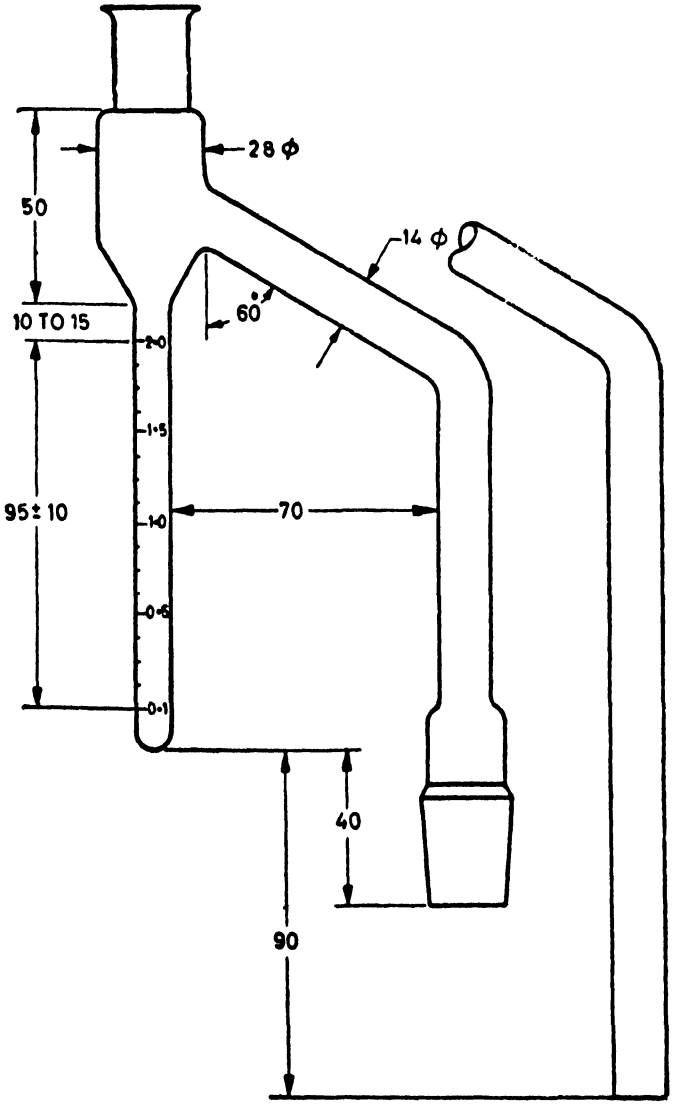
All dimensions in millimetres.

FIG. 3 CONDENSER

TABLE 2 MANDATORY DIMENSIONS AND TOLERANCES FOR RECEIVER

(*Clauses* B-1.1.3.1 *and* B-1.1.3.4)

SL No.	CHARACTERISTIC	DIMENSION
(1)	(2)	(3)
i)	Volume equivalent to smallest sub-division, ml	0·05
ii)	Scale length, mm	95 ± 10
iii)	Length of cylindrical tube above upper graduation mark, mm	10 to 15
iv)	Tolerance on capacity, ml	± 0·02



All dimensions in millimetres.

FIG. 4 2-ml RECEIVER

B-1.1.3.5 Each receiver shall have permanently and legibly marked:

a) the abbreviation 'ml',

b) the inscription '27°C' to indicate that the receiver is graduated for content at 27°C, and

c) an identification number shall also appear on the key.

B-1.1.4 *Heat Source* — The source of heat may be either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath shall not be much higher than the boiling point of xylene or toluene, whichever solvent is used.

B-1.1.5 *Copper Wire* — long enough to extend through the condenser, with one end twisted into a spiral. The diameter of the spiral shall be such that it fits snugly within the graduated portion of the receiver and yet may be moved up and down.

B-2. REAGENTS

B-2.1 Potassium Dichromate-Sulphuric Acid Cleaning Solution

B-2.2 Xylene or Toluene — Saturate the xylene or toluene by shaking with a small quantity of water, and distil. Use the distillate for the determination of moisture.

B-3. PROCEDURE

B-3.1 Clean the entire apparatus with potassium dichromate-sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before using.

B-3.2 Place 200 g of the material, accurately weighed, in the distillation flask, add an equal volume of xylene or toluene, as desired, and swirl to mix. Assemble the apparatus and fill the receiver with the solvent by pouring it through the condenser until it begins to overflow into the distillation flask. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture within the tube. In order that the refluxing may be under control, wrap the flask and the tube leading to the receiver with asbestos cloth. Heat the flask so that the distillation rate is about 100 drops per minute. When the greater part of the water has distilled over, increase the distillation rate to about 200 drops per minute and continue until no more water is collected. Purge the reflux condenser occasionally during the distillation with 5-ml portions of xylene or toluene to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to

separate from the xylene or toluene by moving the spiral copper wire up and down in the condenser and receiver occasionally, thus causing the water to settle at the bottom of the receiver. Reflux until the water-level in the receiver remains unchanged for 30 minutes and then shut off the source of heat. Flush the condenser with either xylene or toluene, as required, making use of the spiral copper wire to discharge any moisture droplets. Immerse the receiver in water at about 27°C for at least 15 minutes or until the xylene or toluene layer is clear, and then read the volume of water.

B-4. CALCULATION

B-4.1 Moisture content, percent by mass $= \frac{100\ V\ D}{M}$

where

V = volume in ml of water,

D = relative density of water at the temperature at which the volume of water is read, and

M = mass in g of the material taken for the test.

APPENDIX C

[*Table* 1, *Item* (iv)]

DETERMINATION OF ASH

C-1. PROCEDURE

C-1.1 Weigh accurately about 10 g of powdered material into a clean tared porcelain or silica crucible. Heat gently and then strongly till constant mass is obtained. Cool the crucible in a desiccator and weigh.

C-2. CALCULATION

C-2.1 Calculate and express the mass of ash obtained as percent of the mass of the material taken for the test.

APPENDIX D

[*Table* 1, *Item* (v)]

ACID WASH TEST

D-1. COLOUR STANDARDS

D-1.1 Stock Solution — For this test, the following three stock solutions are required. They are prepared by dissolving the solids mentioned

below in one litre of one percent (*m/m*) hydrochloric acid prepared from acid conforming to IS : 265-1962*:

a) *Red* — 119·0 g of cobalt chloride ($CoCl_2.6H_2O$);

b) *Yellow* — 45·1 g of ferric chloride ($FeCl_3.6H_2O$); and

c) *Blue* — 62·4 g of cupric sulphate ($CuSO_4.5H_2O$).

D-1.2 Preparation — Prepare colour standards by mixing the stock solutions and water in the volumes given against each standard:

Colour Standard No.	*Volume of Stock Solution*			*Volume of Water*
	Red	Yellow	Blue	
(1)	(2)	(3)	(4)	(5)
1	1·5	1·3	1·0	8·2
2	3·6	0·7	1·5	6·2
3	4·8	0·5	2·0	4·7
4	6·0	0·5	1·0	4·5

D-1.2.1 Keep these colour standards in large test tubes, which shall be as nearly identical as possible with the tube used for the sample. Take particular care that the colouration of the glass in all the tubes is the same, and that it is at a minimum.

D-2. PROCEDURE

D-2.1 Take 10 ml of concentrated sulphuric acid (conforming to IS : 266-1961†) in a 150 × 25 mm test tube. Immerse the tube in a hot water-bath till the acid attains the temperature of 80°C. Add 2 g of crushed material into the hot acid and shake gently for exactly 2 minutes, controlling the temperature of the acid at 80°C. Immediately compare the colour thus produced with the colour standards prepared as specified under **D-1.2**.

D-3. REPORT

D-3.1 Report the colour produced as equal to a particular colour standard or, if intermediate between the two standards, as being darker than one and paler than the next standard.

*Specification for hydrochloric acid (*revised*).
†Specification for sulphuric acid (*revised*).

APPENDIX E

[*Table* 1, *Item* (vi)]

DETERMINATION OF MATTER INSOLUBLE IN BENZENE

E-1. REAGENTS

E-1.1 Benzene

E-2. PROCEDURE

E-2.1 Weigh accurately about 10 g of the powdered material. Transfer this to a 500-ml beaker with 250 ml of benzene. Heat to 30 to 40°C over a water bath with constant stirring and allow to settle. Filter through a tared sintered or gooch crucible, wash thoroughly and dry in an oven at 100 ± 2°C. Cool in a desiccator and weigh. Repeat the process of drying and weighing until constant mass is obtained.

E-3. CALCULATION

E-3.1 Matter insoluble in benzene, percent by mass $= \frac{A \times 100}{M}$

where

A = mass in g of the residue, and

M = mass in g of the material taken for the test.

APPENDIX F

[*Table* 1, *Item* (vii)]

DETERMINATION OF NON-VOLATILE MATTER

F-1. PROCEDURE

F-1.1 Weigh accurately about 10 g of the powdered material in a flat-bottomed, circular, porcelain or silica dish and spread the material uniformly in the dish. Heat for about 1 hour in an oven maintained at 160 ± 2°C. Cool in a desiccator and weigh. Repeat the process of drying and weighing till constant mass is obtained.

F-2. CALCULATION

F-2.1 Non-volatile matter, percent by mass $= \frac{A \times 100}{M}$

where

A = mass in g of the residue left, and

M = mass in g of the material taken for the test.

APPENDIX G

(*Clause* 6.1)

SAMPLING OF NAPHTHALENE

G-1. GENERAL REQUIREMENTS OF SAMPLING

G-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

G-1.1 Samples shall not be taken in an exposed place.

G-1.2 The sampling instrument shall be clean and dry.

G-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

G-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

G-1.5 The samples shall be placed in suitable clean, dry and air-tight glass containers on which the material has no action.

G-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

G-1.7 Each sample container shall be sealed air-tight with a glass stopper after filling and marked with full identification particulars such as sample number, the date of sampling, grade and batch of manufacture of the material and other important particulars of the consignment.

G-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

G-2. SCALE OF SAMPLING

G-2.1 Lot — In a single consignment, all the packages of the same size and containing material of the same grade and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of packages of different sizes or containing material of different grades or batches of manufacture, then the packages of the same size containing material of the same grade and batch of manufacture shall be grouped together and each group shall constitute a separate lot.

G-2.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of packages to be selected for drawing the samples shall depend upon the size (N) of the lot and shall be in accordance with Table 3.

TABLE 3 SCALE OF SAMPLING

No. of Packages in the Lot (N)	No. of Packages to be Selected (n)
Up to 15	3
16 to 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

G-2.3 The packages shall be selected at random. In order to ensure the randomness of selection, random sampling procedures given in IS : 4905-1968* shall be followed.

G-3. PREPARATION OF SAMPLES

G-3.1 From each of the packages selected in **G-2.2**, draw with an appropriate sampling instrument small portions of the material from different parts of the package. The material so drawn from a package shall be about 1 kg. This shall be the representative sample for the package.

G-3.2 From the samples representing different packages selected in **G-2.2**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient to carry out triplicate tests for the characteristics specified under **G-4.2**. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier, and the third for the referee.

*Methods for random sampling.

G-3.3 The remaining portions of the material in samples from each package shall be divided into three equal parts, each forming an individual sample (*see* **G-4.1**). One set of individual samples representing the *n* packages selected shall be for the purchaser, another for the supplier and the third for the referee.

G-3.4 All the individual and composite samples shall be transferred to separate containers. These sample containers shall then be sealed with stoppers and labelled with full identification particulars as given in **G-1.7**.

G-3.5 The referee sample consisting of the composite sample and set of *n* individual samples shall bear the seales of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in the case of any dispute between the two.

G-4. NUMBER OF TESTS

G-4.1 Tests for the determination of crystallizing point and moisture content shall be conducted on each of the individual samples.

G-4.2 Tests for the determination of all the remaining characteristics shall be conducted on the composite sample.

G-5. CRITERIA FOR CONFORMITY

G-5.1 For Individual Samples — The lot shall be declared as conforming to the requirements of crystallizing point and moisture content if each of the individual test results satisfies the relevant requirements given in Table 1.

G-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all the other characteristics determined on the composite sample the test results for each of the characteristics shall satisfy the relevant requirements given in the specification.

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